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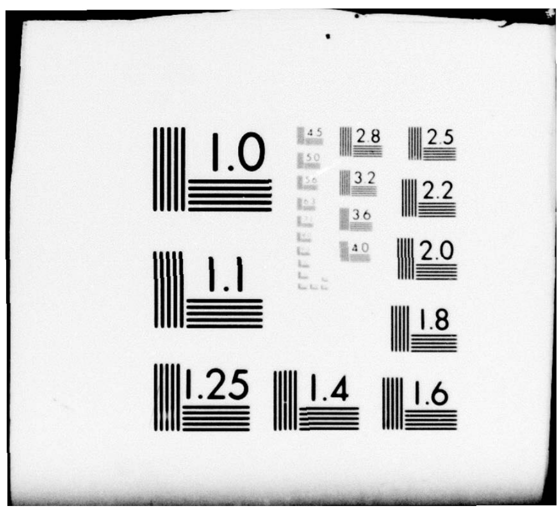
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TECHNICAL REPORT ARBRL-TR-02204

(Supersedes IMR No. 598)

A METHOD FOR THE DETERMINATION OF THERMAL CONDUCTIVITY OF PROPELLANT MATERIALS BY DIFFERENTIAL SCANNING CALORIMETRY

Warren W. Hillstrom

December 1979

US ARMY ARMAMENT RESEARCH AND DEVELOPMENT COMMAND
BALLISTIC RESEARCH LABORATORY
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This report supersedes Interim Memorandum Report No. 598.

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Thermal conductivity	Differential scanning colorimetry
Thermal analysis	Heat transfer
Propellants	

20. ABSTRACT (Continue on reverse side if necessary and identify by block number) (mba)

Benzoic acid, polymethylmethacrylate, polytetrafluoroethylene, and X-14 (a high energy, double-base propellant) were heated through decomposition and their thermal analysis curves compared. X-14 undergoes a pyrolytic decomposition beginning at 413°K and peaking at 573°K. The thermal conductivities of small samples of propellant and polymer were calculated from measurements of their rate of heat flow into a heat sink in a modified Differential Scanning Calorimeter. This method will permit thermal conductivity measurements in small

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samples of sensitive materials, thus reducing hazards in their handling, and giving the first measurement on some very sensitive materials.

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I. INTRODUCTION

The thermal conductivity of materials are commonly determined experimentally by heating relatively large samples for hours or days in each determination. The large sample size and long heating periods greatly increase the hazards involved if the samples are explosives or propellants. However, thermal characteristics such as thermal conductivity of such materials are critically needed in order to model their ignition and combustion.

Thermal analysis presents an opportunity to determine thermal characteristics of explosives and propellants using very small samples of material[1]. The object of this work was to measure the heat flow rate through polymeric and propellant samples using a Thermal Analyzer with a Differential Scanning Colorimeter (DSC) and from this to calculate their thermal conductivities. In addition, the exothermicity or endothermicity of the materials were also to be measured by Differential Thermal Analysis (DTA). Such thermal events in energetic materials indicate condensed phase chemical reactions such as thermal decomposition or physical transformations which precede ignition.

rest of doc refers to it as Calorimeter

In DTA the sample and reference are heated in a furnace at some preset linear heating rate. The furnace temperature and the difference in temperature between the sample and reference materials are displayed on the Thermal Analyzer. If the sample temperature increases faster than the reference temperature, an exothermic change is occuring and heat is given off during the process. If the sample temperature increases slower, an endothermic change is occurring and heat is absorbed in the process. The DSC is somewhat similar to the DTA except that the sample and reference are heated through a constantan disc which not only supports them, but also serves as one element of the temperature measuring thermoelectric junctions. Since the mode of heat transfer is reproducible for a given atmosphere and the thermocouple is not in the sample, the ordinate value of a thermogram at any given temperature is directly proportional to the differential heat flow between the sample and reference materials. This allows quantitative measurement of thermal occurrences.

An equation may be derived[2] from the Fourier equation of heat flux to calculate thermal conductivity from heat flow in a DSC.

---

[1]*P. D. Garn, "Thermoanalytical Methods of Investigation," Academic Press, New York, 1965.*

[2]*F. N. Larsen and C. L. Long, 26th Pittsburgh Conference on "Analytical Chemistry and Applied Spectroscopy," Cleveland, Ohio, 1975.*

$$k_T = \frac{E_T\,(S)(L)(\Delta y)(T_1 - T_2)}{A\,(T_1 - T_3)} \qquad (1)$$

where

k_T = Thermal conductivity at test temperature (mWatt/cm °C),

E_T = Calibration coefficient of the DSC (mWatt/mV),

S = Slope of heat flow versus temperature curve at test temp (cm/°C),

L = Thickness of sample (cm),

A = Area of sample (cm^2),

Δy = Y-axis sensitivity (mV/cm),

T_1 = Temperature at base of sample at test temperature,

T_2 = Temperature at base of sample at start of run, and

T_3 = Temperature of heat sink at top of sample at test temperature.

II. EXPERIMENTAL APPARATUS AND MATERIALS

The DuPont 900 Thermal Analyzer was used with a DTA cell and the DuPont 990 Thermal Analyzer was used with a DSC cell. The DTA were done with 4mm columns of powder in 2mm diameter sample tubes. DSC measurements for calibration of the heat flux were done with a solid sapphire disc directly on the constantan platform without a reference.

For thermal conductivity measurements the DSC cell was used as shown in Figure 1. An insulator was placed over the constantan disc with an opening directly over the sample. The insulator was constructed from a machinable, ceramic-like material, Plastonium C-D, supplied by Insulation Systems, Inc., Santa Ana, California. The heat sink and a rod connecting it with the top of the sample were constructed from 99.9% purity, hard temper, deoxidized copper (Federal Specification QQC-503). A nitrogen flow blanketed the samples at 8 cm^3/sec.

The cylindrical samples were in general 5mm diameter and 4mm length, but each sample was measured accurately for calculation of the thermal conductivity. Polytetrafluoroethylene (Teflon) and polymethylmethacrylate (Plexiglass) were obtained locally. The specific gravities of the materials were measured to characterize them. The polymethylmethacrylate was 1.17g/cm^3. The polytetrafluoroethylene was 2.16 g/cm^3.

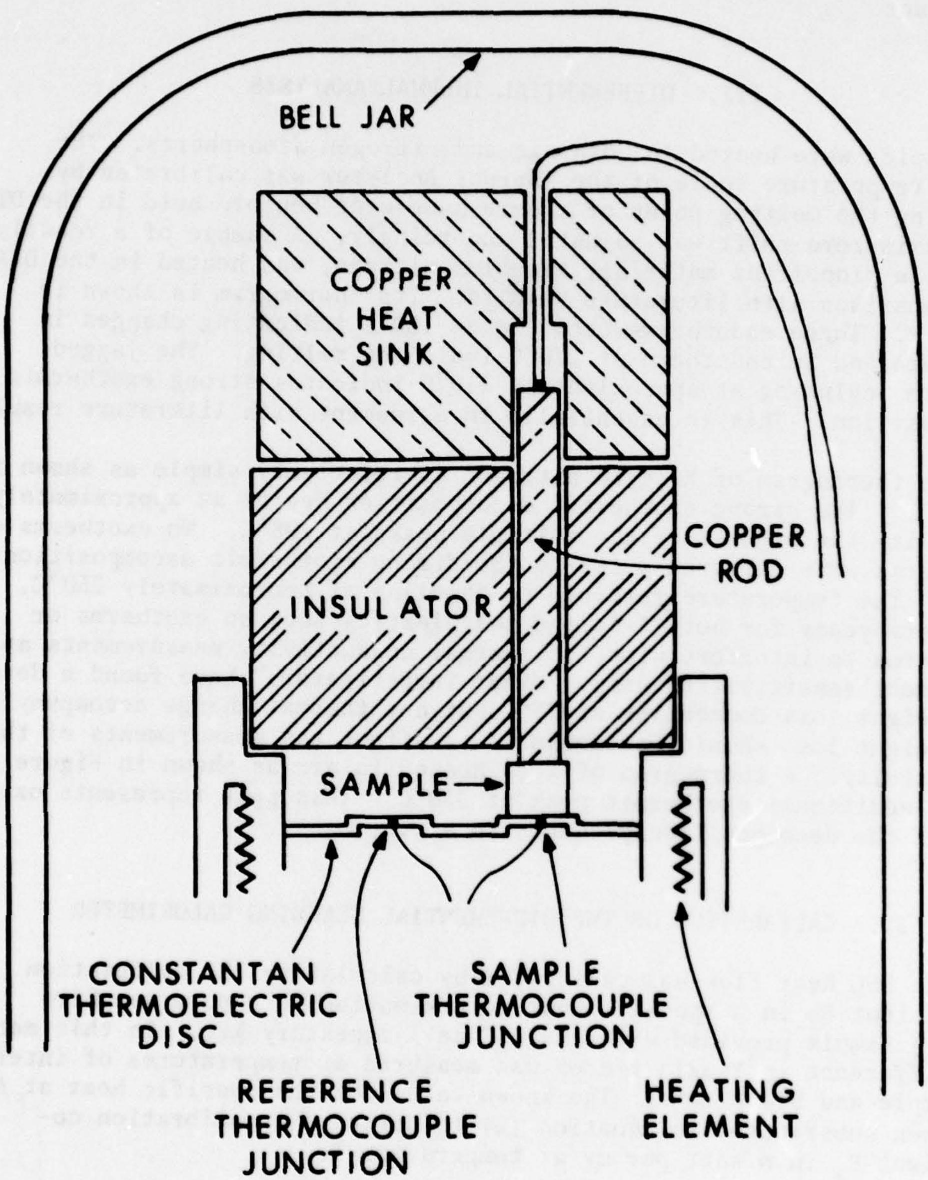

Figure 1. Differential scanning calorimeter modified for thermal conductivity measurements.

Both correspond to literature values. The X-14 propellant was obtained by J. R. Ward of the Ballistic Research Laboratory from the Naval Ordnance Laboratory, White Oak, MD. X-14 is a high energy, double-base propellant.

III. DIFFERENTIAL THERMAL ANALYSIS

Samples were heated in both air and nitrogen atmospheres. The x-axis temperature scale of the Thermal Analyzer was calibrated by measuring the melting point of a pure sample of benzoic acid in the DTA. The x-axis zero shift was adjusted accordingly. A sample of a readily available propellant material, ammonium nitrate, was heated in the DTA for comparison with literature results. Its thermogram is shown in Figure 2. Three endotherms occur below 150°C indicating changes in structure and an endotherm at 170°C indicates melting. The jagged exotherm beginning at approximately 210°C indicates strong exothermic decomposition. This is essentially in agreement with literature results[3].

The thermogram of X-14 in nitrogen is relatively simple as shown in Figure 3. The strong exothermic decomposition begins at approximately 140°C with the highest of the multiple peaks at 198°C. No exotherms or endotherms were detected prior to the strong exothermic decomposition peaks. The temperature returned to baseline at approximately 250°C. DSC thermograms for both X-14 and the plastics show no exotherms or endotherms to interfere with the thermal conductivity measurements at the instrument sensitivities used. Other investigators[4] have found a detectable weight loss commencing at 75°C but any thermal change accompanying this weight loss should be too small to affect the measurements of thermal conductivity. A thermogram of X-14 heated in air as shown in Figure 4 has an additional exothermic peak at 339°C. This peak represents oxidation of the decomposition products in air.

IV. CALIBRATION OF THE DIFFERENTIAL SCANNING CALORIMETER

The DSC heat flow was calibrated by calculating the calibration coefficient E_T in a specific heat determination on a pure sapphire ($A\ell_2O_3$) sample provided with the DSC cell accessory kit. In this method the difference in Y-axis traces was measured at temperatures of interest in sample and blank runs. The known value[5] of the specific heat at $A\ell_2O_3$ was then substituted in Equation (2) to derive the calibration coefficient E_T in m Watt per mv at temperature T.

---

[3] *E. I. DuPont deNemours & Co. (Inc.) Instruction Manual, 900 Thermal Analyzer and Modules, Wilmington, Del., 1968.*

[4] *J. R. Ward, Anal. Colorimetry* <u>*4*</u> *143 (1977).*

[5] *D. C. Ginnings and G. T. Furukawa, J. Am. Chem. Soc* <u>*75*</u> *522 (1953).*

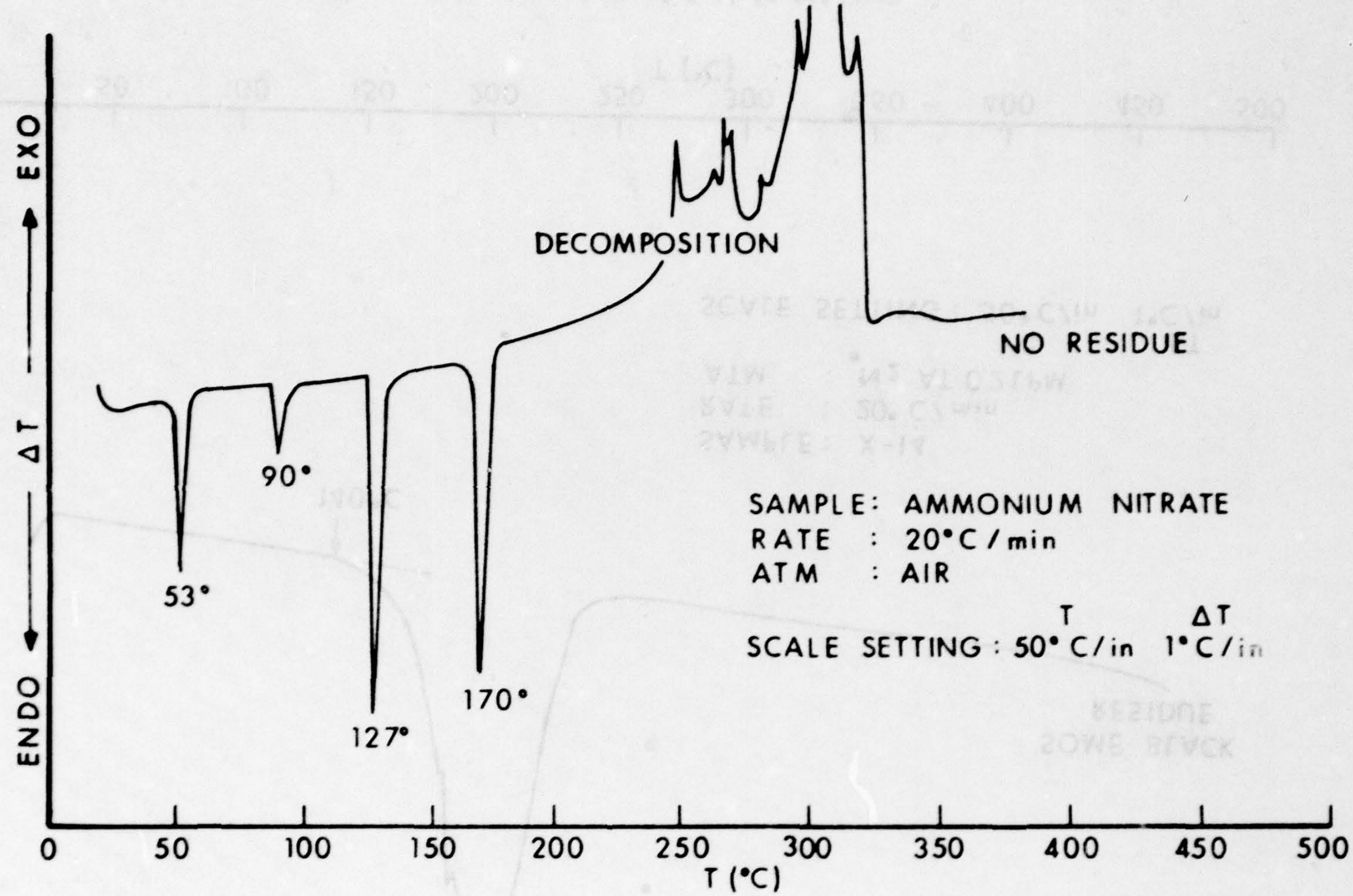

Figure 2. DTA thermogram of ammonium nitrate in air.

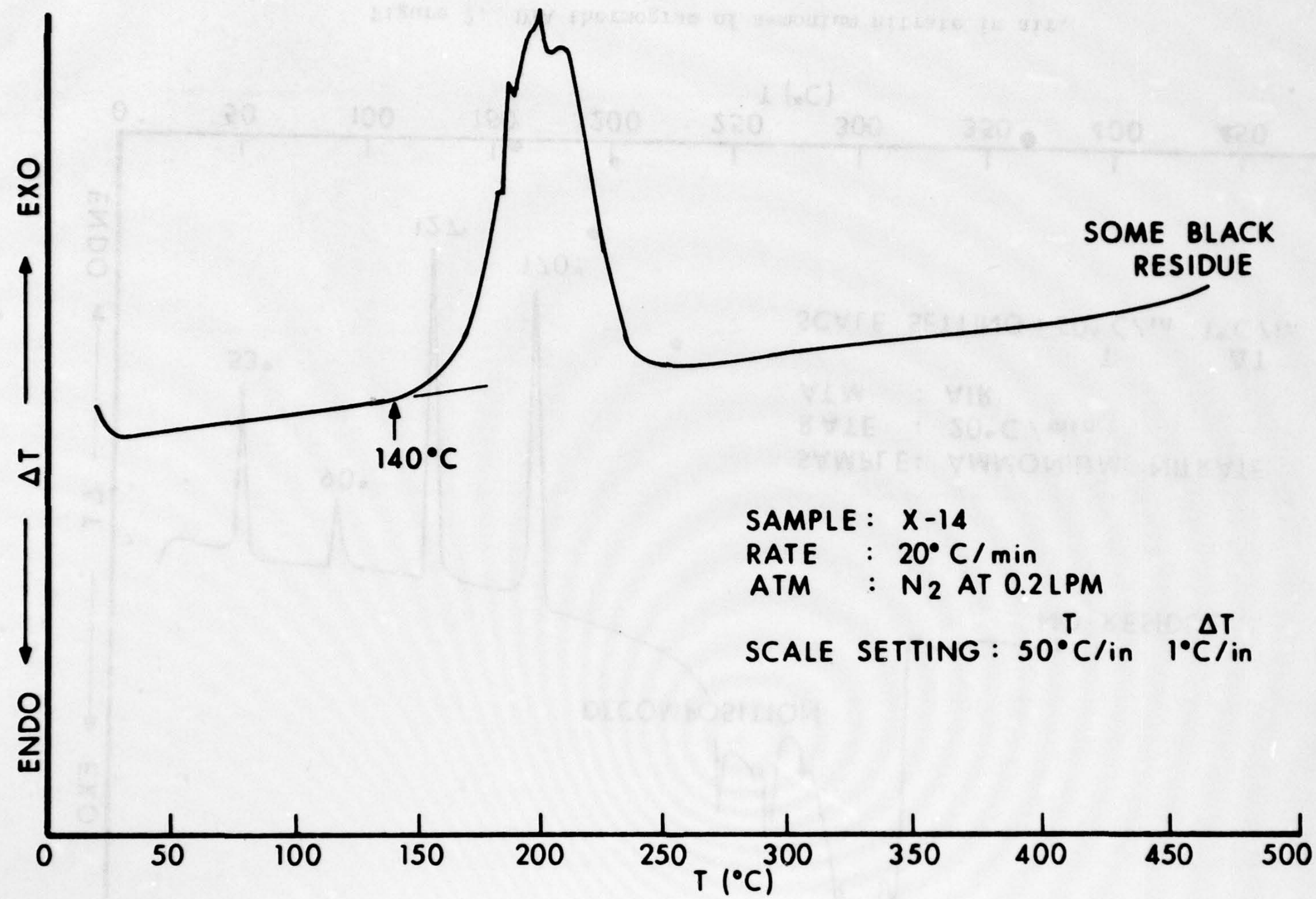

Figure 3. DTA thermogram of X-14 in nitrogen.

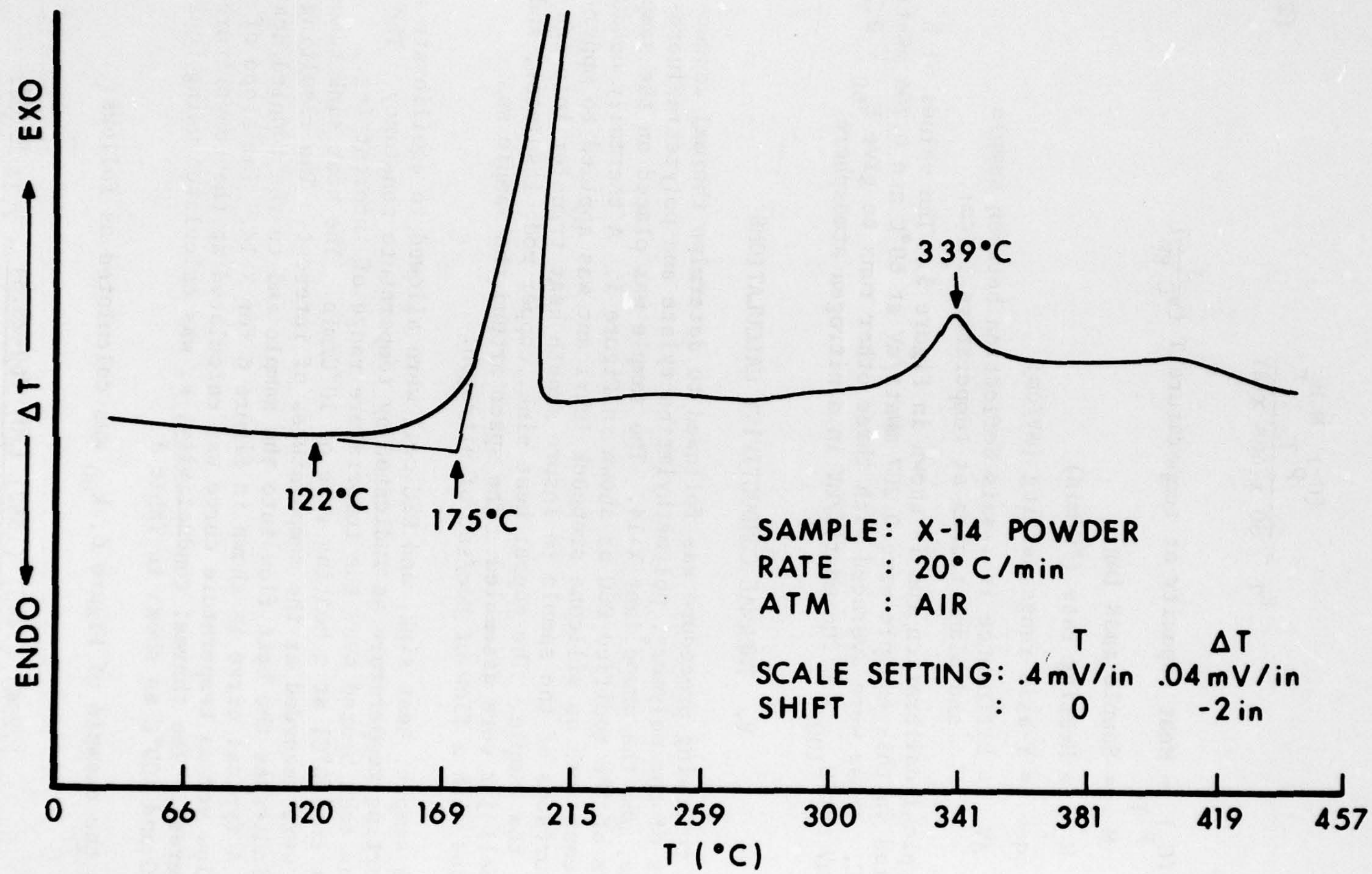

Figure 4. DTA thermogram of X-14 in air.

$$E_T = \frac{(C_p)_T \; M \; H_r}{60 \times \Delta qs \times \Delta Y} \qquad (2)$$

where

$(C_p)_T$ = Heat capacity at temperature T $(\frac{J}{°C\ gm})$

M = Sample mass (mg)

H_r = Heating rate (°C/min)

Δqs = Y-axis range setting (mV/cm)

ΔY = Difference in Y-axis deflection between sample and blank traces at temperature T (cm).

A typical calibration run is shown in Figure 5. The values of E calculated in this example were 0.207 mWatt/mV at 60°C and 0.206 mWatt/mV at 100°C. These were averaged with three other runs to give E_{60} = 0.206 mWatts/mV and E_{100} = 0.206 mWatts/mV in a nitrogen atmosphere.

V. THERMAL CONDUCTIVITY CALCULATIONS

The following procedure was followed to determine thermal conductivities of the polymers, polymethylmethacrylate and polytetrafluoroethylene, and the propellant X-14. The sample was placed on the sample platform of the modified DSC as shown in Figure 1. A thermally conductive grease such as silicone stopcock lubricant was applied to upper and lower surfaces of the sample to insure smooth heat transfer into and through the sample. The copper heat sink, copper rod, insulator, and glass bell jar were assembled. The space around the sample was blanketed with a flow of 8cc/sec of nitrogen.

The sample, heat sink, and DSC cell were allowed to equilibrate at the starting temperature as indicated by temperature constancy. The DSC cell was then heated over the temperature range of interest (eg., ambient to 100°C) at a heating rate of 10°C/min. The heat sink temperatures were recorded at the temperatures of interest. The resulting curve indicates the heat flow into the sample and to the coupled heat sink. A typical curve is shown in Figure 6 for X-14. The slope of the heat flow versus temperature curve was calculated at the temperatures of interest. The thermal conductivity, k, was calculated using Equation 1 at 60 and 100°C as shown in Table I.

In the example of Figure 6, k_{60} was calculated as follows:

$$k_{60} = \frac{(.206)\ (.445)\ (.382)\ (10)\ (60\text{-}25.5)}{(.157)\ (60\text{-}24.0)} = 2.14\ \frac{\text{mWatt}}{\text{cm°K}}$$

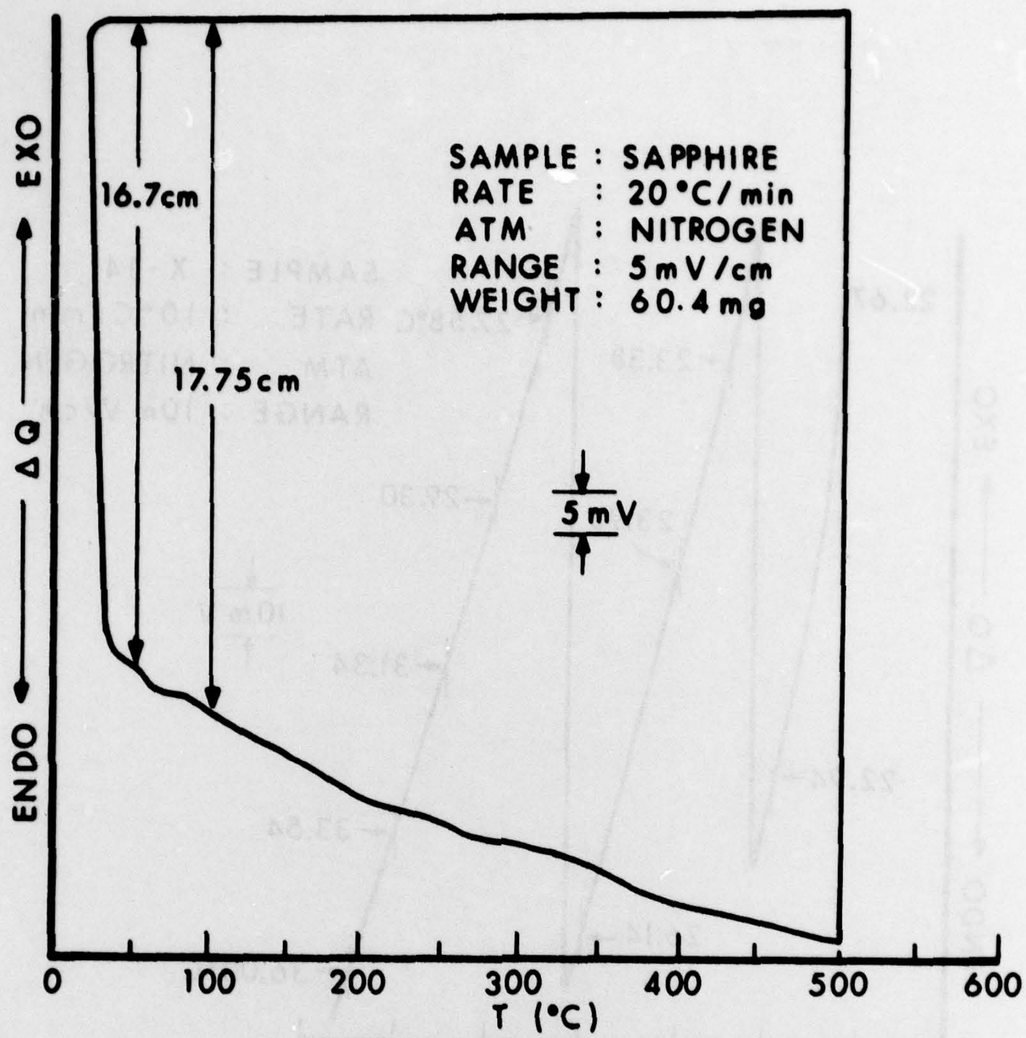

Figure 5. Specific heat determination of sapphire.

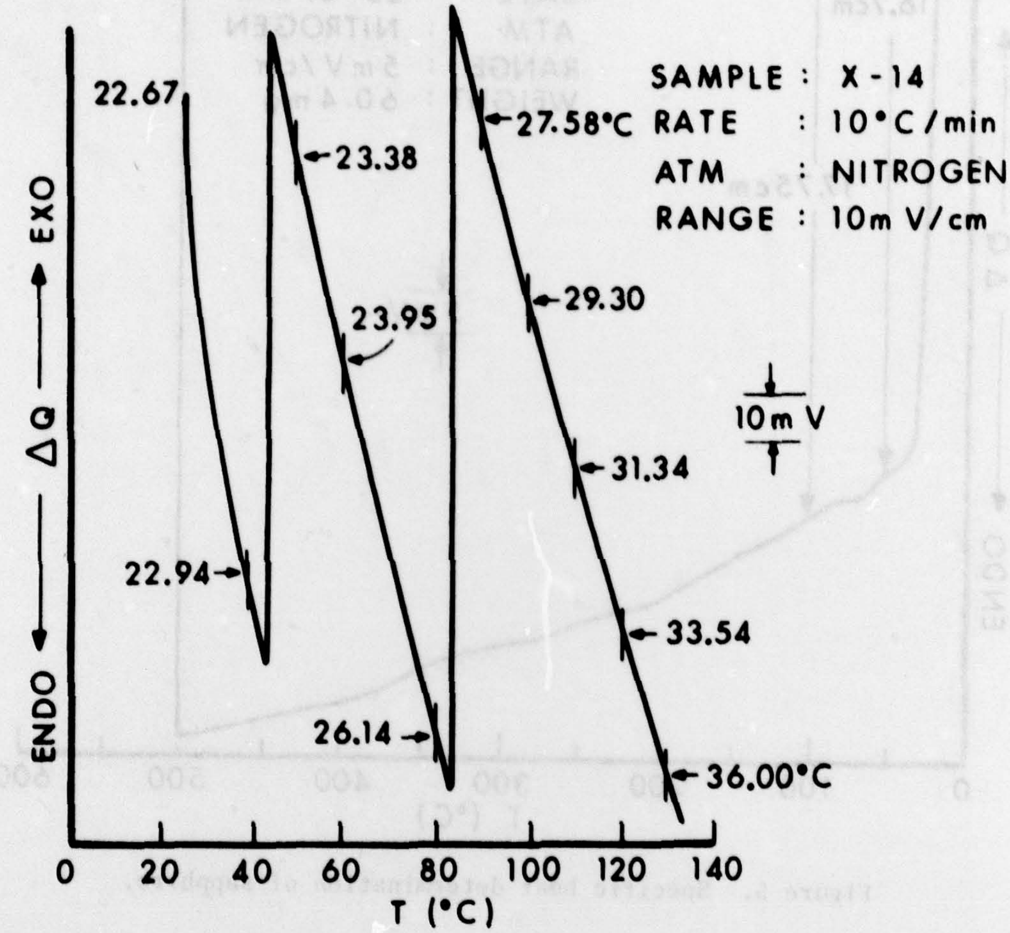

Figure 6. Thermal conductivity determination on Polymethylmethacrylate.

Table I. Thermal Conductivity Characteristics

Run No.	Sample Material*	Sample Length cm	Sample Area cm^2	Slope at 60°C cm/°C	Top Temp at 60°C	k_{60}**	Slope at 100°C	Top Temp at 100°C	k_{100}**
1	PMM	0.447	0.168	0.615	26.5	1.61	0.575	31.3	1.65
2	PMM	0.447	0.168	0.315	26.5	1.65	0.285	31.2	1.64
3	PMM	0.447	0.168	0.310	27.4	1.64	0.280	31.7	1.61
4	PMM	0.444	0.168	0.300	24.3	1.51	0.285	29.5	1.61
5	PMM	0.444	0.168	0.295	24.5	1.47	0.265	28.0	1.45
6	PMM	0.457	0.168	0.290	25.4	1.55	0.270	30.3	1.58
7	PTE	0.376	0.168	0.490	24.9	2.12	0.438	30.4	2.12
8	PTE	0.450	0.168	0.429	27.0	2.29	0.380	31.8	2.21
9	PTE	0.450	0.168	0.385	24.8	2.02	0.358	29.8	2.07
10	PTE	0.450	0.168	0.433	24.9	2.28	0.395	30.3	2.30
11	PTE	0.452	0.168	0.415	25.2	2.21	0.375	30.4	2.20
12	X-14	0.382	0.157	0.445	24.0	2.14	0.388	29.3	2.05
13	X-14	0.378	0.157	0.445	26.2	2.12	0.388	31.4	2.03
14	X-14	0.378	0.148	0.443	26.1	2.20	0.390	31.1	2.14

* PMM = Polymethylmethacrylate and PTE = Polytetrafluoroethylene

** k_T in mWatt/cm°K

The thermal conductivities were averaged for each material to give Table II.

Table II. Thermal Conductivity

Sample Material	k60 mWatt/cm°K	k100 mWatt/cm°K
Polymethylmethacrylate	1.57	1.59
X-14	2.15	2.07
Polytetrafluoroethylene	2.18	2.18

The thermal conductivities measured by this DSC technique for the two polymers are in good agreement with literature values. Thus, Lucks[6] measured the thermal conductivity of polymethylmethacrylate to be 1.54×10^{-3} W/cm °K at 27.1°C and 1.57×10^{-3} W/cm °K at 59.0°C by a longitudinal heat flow method. Krischner and Esdorn[7] measured the polymethylmethacrylate thermal conductivity to be 1.92×10^{-3} W/cm °K at 25°C (298°K) using a transient heat flow method which has greater possibility of error than the longitudinal heat flow method. Larsen and Long[2] measured the thermal conductivity of polytetrafluoroethylene to be 1.80 mWatt/cm°K with no temperature given. Schultz and Wong[8] measured thermal conductivities of 4.01×10^{-3} W/cm °K for the same material at 166°C (439.3°K).

The trend of decreasing thermal conductivity with increasing temperature for the X-14 propellant seen in Table II is somewhat different from that of the polymers, but such a trend has been observed for a number of explosives such as PBX-9404.[9] A value of 2.30×10^{-3} W/cm °K is reported[9] for nitrocellulose (12% N) with no temperature given. Thus, both the numerical value and effect of temperature on thermal conductivity measured for the propellant are consistent with literature values.

[6] *C. F. Lucks, G. F. Bing, J. Matolich, H. W. Deem, and H. B. Thompson, "The Experimental Measurement of Thermal Conductivities, Specific Heats, and Densities of Metallic, Transparent, and Protective Materials," USAF TR 6145 (1952), AD 95239.*

[7] *O. Krischner and H. Esdorn, VDI Forschungshelf 450 Suppl. to Forsch. Gebiete Ingenieurw., B. (21) 28-39 (1955) in "Thermal Conductivity of Non-Metallic Solids," Ed. by Y. S. Touloukian, 1970, IFI/plenum.*

[8] *A. W. Schultz and A. K. Wong, "Thermal Conductivity of Teflon, Del-F, and Duroid 5600 at Elevated Temperatures," Watertown Arsenal Laboratories Technical Report 397/10, (1958), AD-154351.*

[9] *B. M. Dobratz, "Properties of Chemical Explosives and Explosive Simulants," UCRL-51319, Rev. 1, July 1974.*

VI. CONCLUSION

The Differential Scanning Calorimeter method gives thermal conductivity determinations comparable with literature results using small sample sizes and short heating periods which are readily applicable to energetic materials such as explosives and propellants.

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